Service Life Prediction Program for Sealant Formulations

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In Modern Architecture, Sealant and Adhesives are Critical for Environmental Integrity

Guggenheim Museum in Bilbao, Spain

Sealant and Adhesive

- Critical to modern building design
- 80% of assets are in constructed facilities
- Sealant and Adhesive materials cost is a small fraction of the total construction cost, but failure results in significant damage and repair cost.
- Failure of sealant is one of the most cited complaints in constructed facilities. “The windows leak” (NAHB)
- Current test methodologies do not allow for accurate prediction of service life, hence progress in increasing durability or decreasing time to market is much more difficult
- Other industries have seen a revolution, mm tolerance program in autos, airframes, in the way they do business. This may be coming to constructed facilities.
- NIST is developing better tools to asses durability of sealant and adhesive.
- These tools may become part of a new rating system for sealant and adhesive.

Who is NIST?

NIST is a non-regulatory federal agency within the Technology Administration of the U.S. Department of Commerce. NIST's primary mission is to ***promote economic growth by working with industry to develop and apply technology, measurements, and standards,*** and to maintain the Nation's measurements and standards infrastructure

NIST carries out its mission through four integrated programs:

Measurements and Standards Program

Advanced Technology Program

Manufacturing Extension Partnership Program

National Quality Program (Baldrige Award)

1901-2001 NIST CENTENNIAL

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3,300 Scientists, Engineers
1,500 Visiting Scientists

Established in 1901
1998 Nobel Prize in Physics

Four Missions:

1) World Leading Metrology- Standards (NIST traceable)
2) Increase Economic Competitiveness & Safety(American Industry)
3) Respond Quickly to Pressing National Needs
4) Facilitate Technical Innovation-ATP

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Service Life Prediction

Current Reality:

Current methodology relies on one of two methods:

Outdoor exposure or **Laboratory exposure**

" This brings us face-to-face with one of the most perplexing problems concerned with outdoor weathering, that the weather does not duplicate itself. How can one ever expect a laboratory method to duplicate the weather when the weather can never duplicate itself" [Grinsfelder, 1967]

" Successful laboratory simulation of the effects of weather on coatings, plastics and other materials has eluded scientists for over fifty years. Published literature report hundreds of attempts to duplicate and accelerate weathering effects and conclude that there is no substitute for natural weathering [Dreger, 1973]

"Current estimates of Service Life Prediction are Crude and there is Little or no Correlation between Laboratory and Field Exposure." Rilem State of the art Report, 1999.

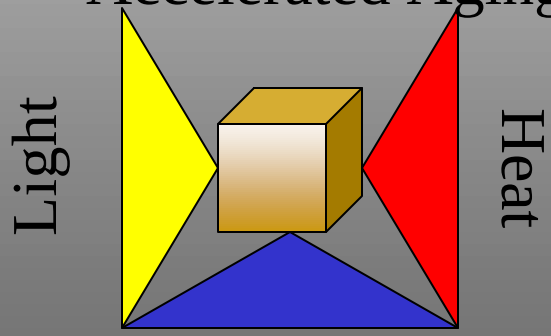
Currently, the Standard is Outdoor Weathering.

Conventional SLP Metrology

Outdoor Aging

Make Comparison
Outdoor
Vs.
Accelerated Aging

Accelerated Aging



No Correlation;
Adjust Accelerated
Again Factors.

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Conventional Methodology: Outdoor Exposure is the Standard of Performance

- Manufactures Dilemma:

Increase Time to Profit or Increase Liability Exposure

Time to Profit

Coatings > 15 yr

IC Chip ~ 4 months

Increased Liability

Fire Retardant Plywood

Polybutyldiene Pipe

Moisture Resistant Coatings

Automotive Clear Coats

Class Action Lawsuits

(4-6 $B/yr. In warranty costs.)

Outdoor Weathering is Considered: Slow, Expensive, Unreliable

Class Action Lawsuits-

Internet Removes Barriers to Organization, Significantly Increases Scrutiny of Products.

- Can you afford your 50 yr warranty 50 yr Data?
- Can you put your faith in the science not the lawyers?

Please keep your messages on topic (i.e. relating to the peeling paint problem).
Messages Posted

Re: Peeling Paint ('92 Camaro) - Randall Vogt 3/06/2000 (0)
peeling paint - Robert Ramirez 3/03/2000 (0)
'96 Dodge Caravan woes - Jackie 3/03/2000 (0)
92 buick lesabre paint - gary 2/28/2000 (0)
peeling paint - Paul and LuAnne Beckwith 2/26/2000 (1)
Re: peeling paint - Tim Segulin 2/28/2000 (0)
94 Plymouth Voyager Peeling Paint - My Settlement - Keith Francis 2/22/2000 (0)
90 Cherokee Limited(peeling paint,transmission problems,brake problems and more - phillis menschner 2/12/2000 (0)
Chrysler class action lawsuit - Stacy 1/04/100 (18)
Re: Chrysler class action lawsuit - Maston Pruett 2/29/2000 (0)
Re: Chrysler class action lawsuit - Bettye Heinrich 2/15/2000 (0)
Re: Chrysler class action lawsuit - Terri Greene 2/05/2000 (0)
Re: Chrysler class action lawsuit - Mary Ann Ryan 1/30/2000 (0)
Re: Chrysler class action lawsuit re Peeling Paint - Peter Schotting 1/30/2000 (3)
Re: Chrysler class action lawsuit re Peeling Paint - steve 1/31/2000 (2)
Re: Chrysler class action lawsuit re Peeling Paint - martha 2/07/2000 (1)
Re: Chrysler class action lawsuit re Peeling Paint - Ann Marie 2/15/2000 (0)
Re: Chrysler class action lawsuit - Robert Carr 1/10/100 (9)
Re: Chrysler class action lawsuit - mike l. parra 1/16/2000 (0)
Re: Chrysler class action lawsuit - Ray Johnson 1/12/100 (0)
Re: Chrysler class action lawsuit - JACOB MATHEW 1/11/100 (1)
Re: Chrysler class action lawsuit - patrick best 2/09/2000 (0)
Re: Chrysler class action lawsuit - mariea shelton 1/11/100 (4)

Service Life Prediction

NIST Insight:
The way the problem is defined,
outdoor versus laboratory
makes the problem intractable.

Has this problem occurred in other scientific fields?

Biology

How do you think about skin exposure?

- Dose versus Damage.
- 1 hour x no sunscreen = 8 hours with SPF 8.

Two issues:

Reciprocity- Medical Literature over 19 orders of Magnitude

Action Spectra – Well Defined in the Literature.

Biological Cumulative Damage Models

$$D_{total}(t) = \int_0^t \int_{\lambda_{min}}^{\lambda_{max}} E_o(\lambda, t)(1-10^{-A(\lambda)})f(\lambda)d\lambda \text{ dt}$$

- D_{total} (t) = Damage to material.
- λ_{min} and λ_{max} = minimum and maximum photolytically effective wavelengths
- $E_o(\lambda,t)$ = spectral UV irradiance from light source
- $(1\text{-}10^{-A(\lambda)})$ = spectral adsorption of specimen
- $\phi(\lambda)$ = spectral quantum yield of specimen
- $A(\lambda)$ = adsorption at wavelength λ

DOSE

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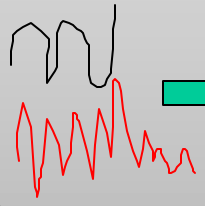
Reliability-Based SLP Methodology

Instrumented Outdoor Exposure

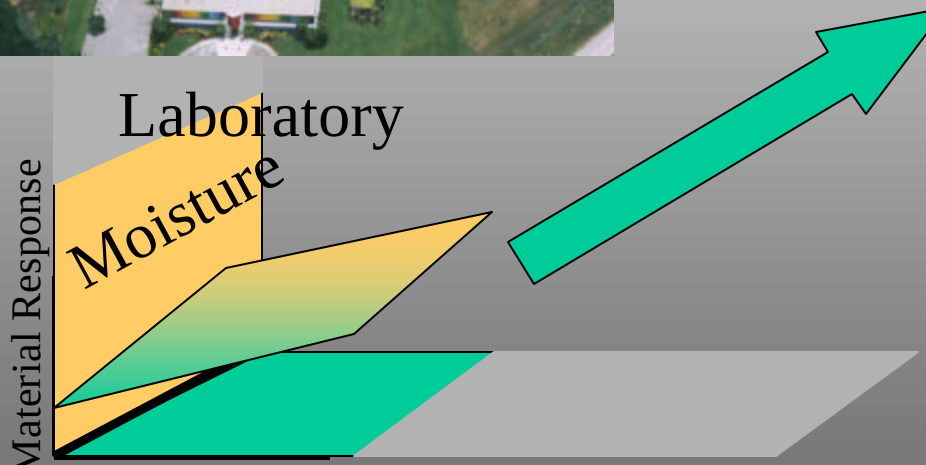
Time Series



Temperature
RH, UV

Cumulative Damage Model

$$D_{total}(t) = \int_0^t \int_{l_{min}}^{l_{max}} E_o(l,t)\left(1-10^{-A(l)}\right) f(l)\, dl\, dt$$



Laboratory

Databases

SLP Estimate

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Example: NIST Coatings Consortia

- Apply Cumulative Damage Models to Organic Coatings.
- Develop metrology to quantify Damage, Dose, Color, Gloss.
- Design Laboratory Exposure Devices that capture and control “real world” conditions:
  - Temperature, Relative Humidity, UV wavelength, UV Dose.
- Instrument Outdoor Exposure Sites
  - Temperature, Relative Humidity, UV wavelength, UV Dose.
- Use data from both Laboratory and Outdoor sites, historical experiments to build database/computer models of SLP.

Coatings Consortia:

- Started 1994, renewed in 1997, 8 companies are currently members.
- New Technologies Created:
  - High Precision Temperature/Humidity Generator.
  - UV exposure device- Intergrating Sphere.
  - Solarnet exposure sites.
- Continued Investment by Industrial Partners.

Why is NIST interested in Sealants?

Housing and Urban Development.

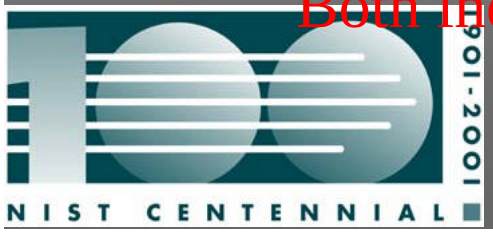
http://www.Pathnet.org/

Partnership for Advancing Technology in Housing – PATH.

80% of Assets are in Constructed Facilities.

The construction industry has not realized the value that programs like mm tolerance program in automotive, airframes have given other industries.

Both industries were spurred by foreign competition.



Path Goals by 2010:

1.AFFORDABILITY

Reduce the monthly cost of new housing by 20 percent or more

2.ENERGY-EFFICIENCY and ENVIRONMENTAL PROTECTION

Cut the environmental impact and energy use of new housing by 50 percent or more and reduce energy use in at least 15 million existing homes by 30 percent or more.

3.DURABILITY

Improve durability and reduce maintenance costs by 50 percent.

4.DISASTER RESISTANCE and SAFETY

Reduce by at least 10 percent the risk of loss of life, injury, and property destruction from natural hazards and decrease by at least 20 percent residential construction work illnesses and injuries.

Durability of Housing Materials and Systems.

- Which materials or systems are the most problematic?

- March 1999, NIST asked NAHB to verify the proposed durability research needs and directions.

- For NIST/PATH-D the report confirmed two serious problematic areas:

Roofing Materials

Sealants

Sealant and Adhesive Program

- Can we adapt the program we have in Service Life Prediction of coatings to Sealant and Adhesive?
- Important Factors in Durability of Sealants (Rilem, ASTM):
  - Temperature, Humidity, UV exposure, Load.
  - Factors must be cyclically interrelated.

Current Durability Standards are Go/No Go

ASTM 3 Test methods:

- C 719 STM for Adhesion and Cohesion of Elastomeric Joint Sealants Under Cyclic Movement. (Hockman Cycle)
- C1442- Standard Practice for conducting Test on Sealant using Artificial Weathering Apparatus
- C 1246- STM for Effect of Heat Aging on Weight Loss, Cracking and Chalking of Sealants after Cure

ASTM- C 719 Hockman Cycle

- Developed at NIST 30 years ago.
- Static Cure of sealant joint 21 days.
- Immersion in water, for 7 days,
- Oven(7 Days)
- Cyclic movement @ room temp.
- Movement at Elevated Temp
- Evaluate with Visual Inspection.

Does this really match in-service life?

Vision:

- Provide a PATH that would eliminate a majority of the weatherproofing problems in Residential housing.

- Metrology for accurate, rapid determination of the durability of new or existing sealant formulations

- Database of sealant durability allows for distinction between products to help guide consumer choice

- Database of sealant durability will serve as an early component for Integrated Decision Support System for Durability in Housing, (Economic Analysis)

How do we get to Reliability Based SLP?

The first thing to do is write a plan.

NES/ASTM E632 Protocol

Elements of a Standard Methodology for Service Life Prediction

- Characterize the service environment
- Characterize the material, component, or system
- Identify the degradation mechanisms
- Develop a model for predicting the rate of degradation
- Define the failure criterion
- Using the model, calculate the time to failure
- Prepare a report of the results in standard format stating clearly the assumptions made

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Industrial Outreach.

NIST

Strengths:

- Resources,
- Equipment,
- Great Metrology
- Focus on Basic Mechanisms
- Neutrality
- International and National Contacts
- Huge Intellectual Resource Pool.

Opportunities:

- Little knowledge about Sealant Formulations, sample design, sample preparation.
- Little Existing Weathering data on Sealants.

Industry

Strengths:

- Great Knowledge of Sealant Formulations of Sealant Formulations
- Existing Weathering Data
- Access to Warranty Data.

Opportunities:

- Ability to gain new exposure tools, protocols.
- Ability to influence new standards development
- Ability to significantly increase durability.

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Relative Roles:

NIST

- Metrology
- Coordination
- Development of Tools
  - Indoor
  - Outdoor
  - Modeling
- Testing
- Verifying
- Standards Development
- Data integrity and access

Industrial Partners

- Sample Selection
- Sample Geometry
- Reservoir of Existing Data
  - Warranty Information
  - In-House Studies
- Depth of Experience

What are the major factors that affect durability?

Important Factors in Durability

What are the Factors that Most Affect Sealant Durability?

From 1999 State of the Art Report: (Rilem/ASTM)

- Moisture/Relative Humidity.
- Temperature
- UV
- Load

The factors must be cyclically interrelated, even during cure.

What tools do we need?

Temperature/Relative Humidity Generator

- Temperature and Relative Humidity Control is of Paramount Importance
- Developed an Apparatus capable of generating four relative humidities between 0% and 90% at one temperature between 25 C and 60 C.
- Temporal Stability
  - Temperature control ± 0.5° C
  - Relative Humidity ± 1 % r.h.

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Product: Novel Integrating Sphere

- 2 M Integrating Sphere Exposure Device
  - High Flux -8,400 W UV radiation.
  - Large sample area- 300-500 samples
  - 95% exposure uniformity between samples
- Visible and Infrared Radiation removed
- UV-radiation broken down into wavebands
- Temperature and relative humidity around specimens well controlled
- Measurements of exposure conditions and degradation response highly automated
- Reasonable cost of equipment relative to exposure area

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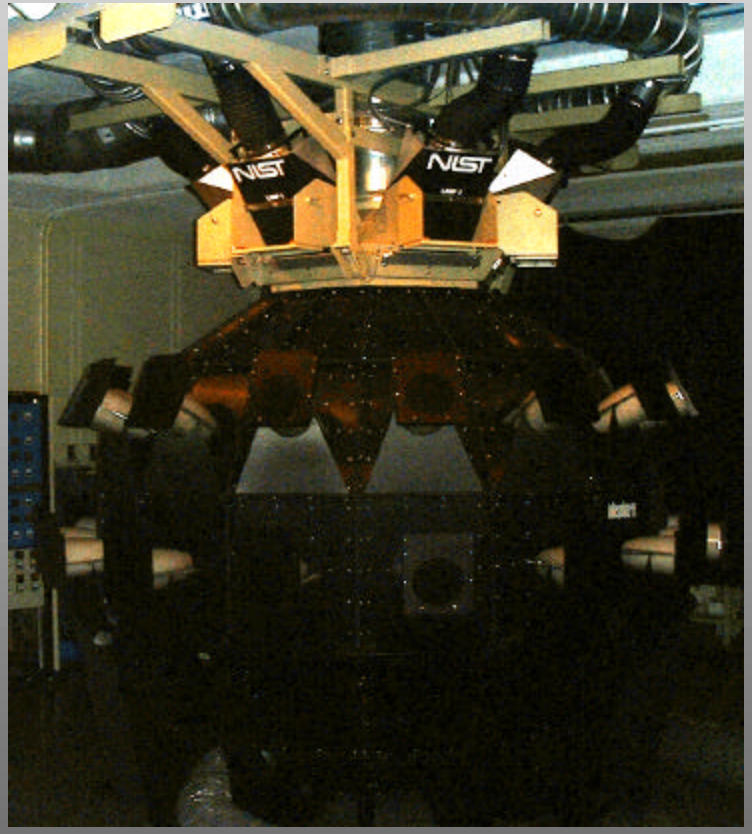
UV Star goes Lights ON!!

Previous

Current.



Methods of Measuring Durability

- Current Method: Visual Inspection
- What Properties are Important for the Seal Integrity?

  The ability of the sealant to accommodate relative motion between two or more supporting surfaces.

- Measure the Rheological Properties.

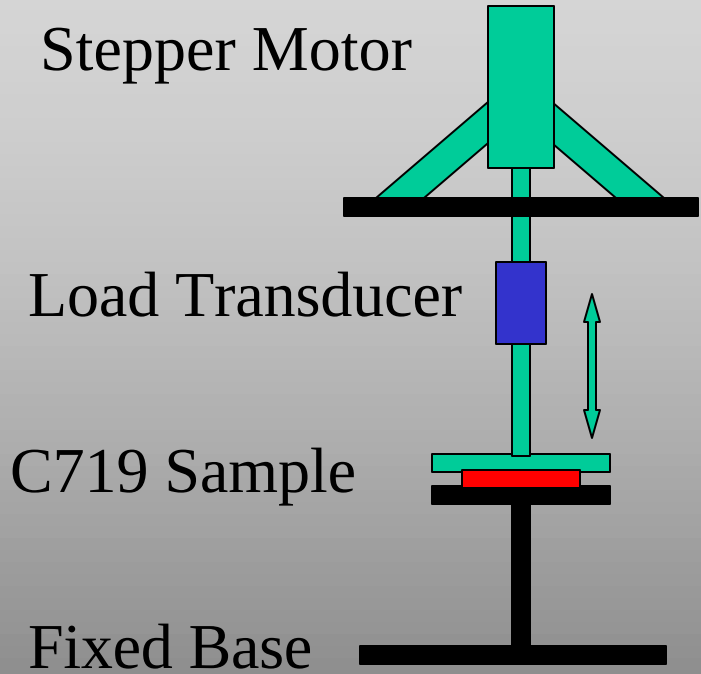
  Produce a known deformation, and measure the force.

  "Stress Strain Curves" or Modulus.

Prototype Sealant Tester

Produce Known Deformation
Measure the Force

$$\frac{\text{Stress}}{\text{Strain}} = \text{Modulus}$$



What's New?

ASTM C719 relies on forensic visual inspection.

This device will measure the performance of the sealant continuously, allowing for detailed description of the failure mechanism.

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Design Apparatus and Initiate Experiments on Sealants

- Device has been assembled
- Evaluation of the device has begun.
- Exciting new capability in the evaluation of performance, every cycle, every sample.
- The trips to Dow Corning, DAP assist in sample preparation issues.
- First issue to be examined will be the effect of movement during cure.

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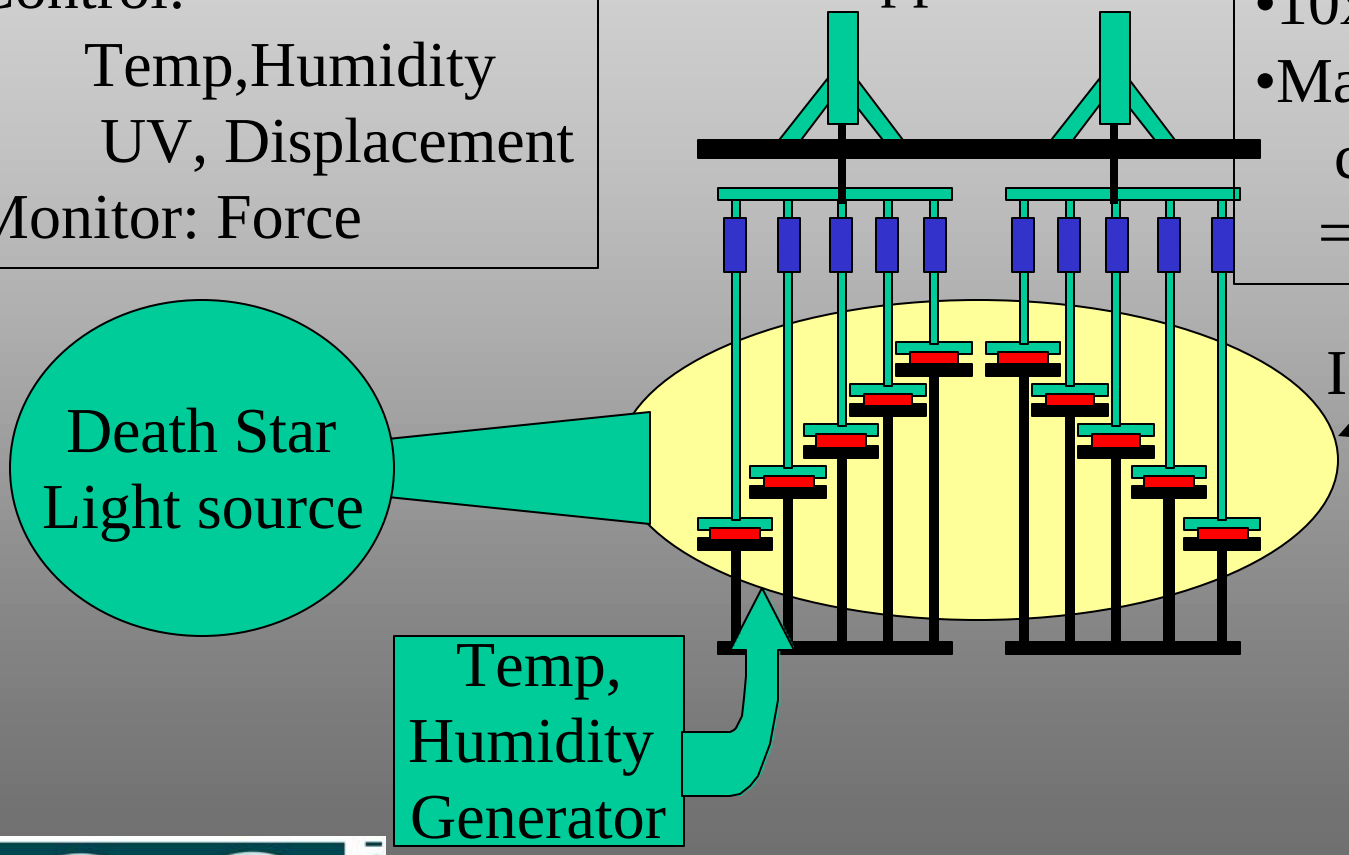
Proposed Full Scale Sealant Tester

Use Computer to Control:
Temp,Humidity
UV, Displacement
Monitor: Force

•30-50 Samples
•10x aging.
•May be 4 separate chambers.
= 200 samples



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Service Life Prediction

Indoor Tools

Humidity Generators
Temperature Controllers
UV Exposure
Motion Controllers
Force Monitoring
Sample Holders
Cyclically Interrelated

Outdoor Tools

Humidity
Temperature
UV Monitors
Sample Holders

Modeling Tools

Data Base
- Organization
- Storage
- Analysis

Predicative Tools

Acceleration Factors

How do we Accelerate the Aging?

Temperature	Raise the Temperature.
Deformation	Increase the Deformation- Superposition
UV	Increase the UV- Cumulative Damage
Humidity	What about Humidity?

How do they work in conjunction?

When do these factors change, how long does it take?

Characterize the Material

The factors change at Dawn and at Dusk. What happens:

ΔTemperature	Thermal Measurements
ΔDeformation	Rheological Response
ΔUV	Instantaneously
ΔHumidity	Hyden Moisture Analyzer

The slowest rate of change determines how fast a deformation cycle can be.

Lets say dawn or dusk takes 1 hour.

Acceleration factor of ~10.

What conditions should we use?

Moist

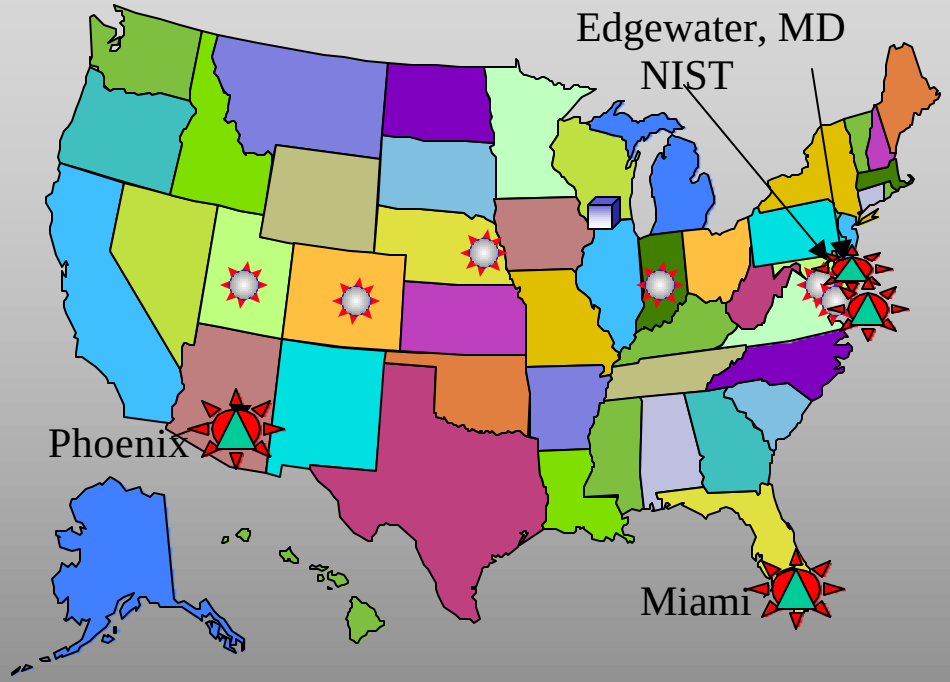
Moist is a database program.

Can Simulate the weather each of the seven major climatic regions identified by NOAA and ASHRAE.

Use this data for the inputs for the control program.

This allows us to realistically simulate the climate (seasonal variation, daily variation) for any one of the monitored cities

Solar Net Exposure Sites:



- 4 sites; spectral solar UV data collected since 1997
- USDA is adding 6 more sites"" to network
- Forest Products Laboratory 2000.
- Corporate Sites?

What is monitored?

- Humidity
- Temperature
- UV (dose, wavelength)
- Sample Holders

FPL

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Additional Comments

There are three factors to increase durability:

- Material Durability — NIST/FPL
- Proper Joint Design — FPL
- Proper Installation — SWRI

Can we integrate these into a "High Performance Sealant?"

High Performance Sealant Rating

Higher Initial Cost:

- Certified Installers
- Proper Joint Design
- Materials that meet a Durability Threshold

Receive:

Economic Analysis of Lifetime Cost Savings Estimate.
Lower Maintenance Costs.
Better Warranty? Guarantee?
Better Joint Performance.

[3.1] Durability Rating System

Round Table (5/26/00)

- Private sector round table at NAHB-RC
- Builders liked the idea of a rating system
- Product manufacturers were divided

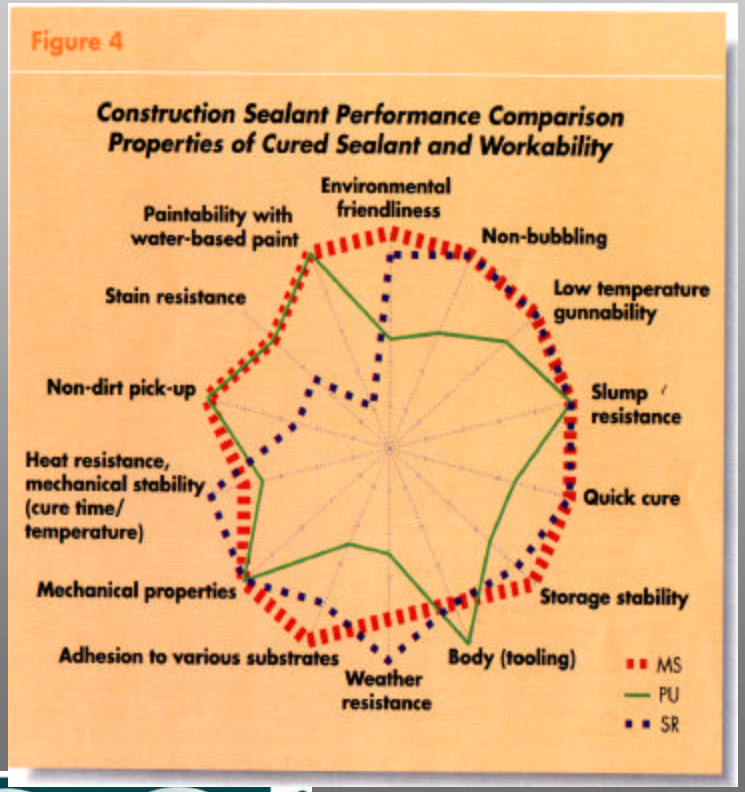
Conclusion and Action

- A prototype multi-attribute product evaluation system will be developed, with joint sealants being featured in the first demonstration

[3.1] Possible rating systems:

An article describing how MS (silyl-terminated polyethers) compares to existing formulations:

*



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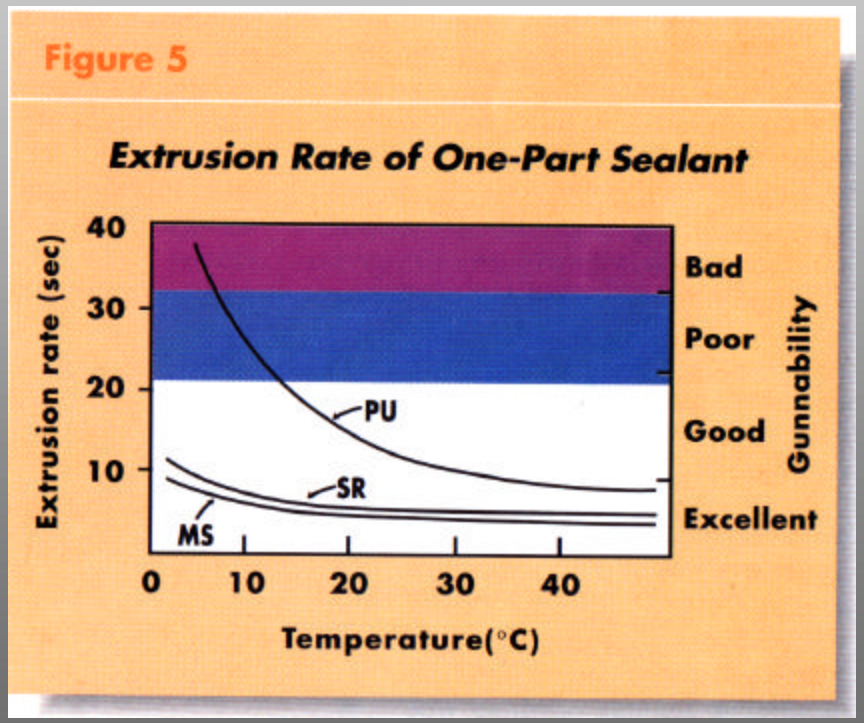

*From Adhesives Age Magazine: 9/98

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[3.2] Decision Support System (I)

- A critical element of the program
- It must:
  - be of the highest integrity
  - be authoritative and well-indexed
  - be interoperable, easily accessible, and user-friendly
  - integrate results from the other PATH-D activities
- BFRL ‘customer’ needs workshop held on June 2
- Prototype system being developed

Vision:

- Provide a PATH that would eliminate a majority of the weatherproofing problems in Residential housing.

- Metrology for accurate, rapid determination of the durability of new or existing sealant formulations

- Database of sealant durability allows for distinction between products to help guide consumer choice

- Database of sealant durability will serve as an early component for Integrated Decision Support System for Durability in Housing, (Economic Analysis)

NIST SWOT

Strengths	Weaknesses
•Industrially Relevant Research •Easy to Defend in Congress	•Lots of Extra Work for Little Payoff •Lots of Voices for Research Direction
Opportunities	**Threats**
• Raise the Bar on Sealant Durability •Significantly Reduce Liability Exposure •Significantly Reduce Building Maintenance Costs. •Significant Advances in Metrology Bringing Science to Weathering.	• Entrenched Test Methods •Perceived Threat to Specific Formulations •"Corporate Welfare" •Change

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Industry SWOT

Strengths	Weaknesses
•Leveraging Federal Resources •People •Funding 10(fed):1(Industry) •Equipment •Ability to Influence Future Standards Development	• Perception: Not relevant to our needs. •Perception: Lots of extra work for little payoff
Opportunities	**Threats**
• **Shorter Product Development Cycles** • Raise the Bar on Sealant Durability •Significantly Reduce Liability Exposure •Significantly Reduce Building Maintenance Costs. •.Eliminate Duplication of Efforts •Level Playing Field.	• Entrenched Test Methods •Perceived Threat to Specific Formulations •Perceived loss of Confidentiality of Proprietary Information •Level the Playing Field

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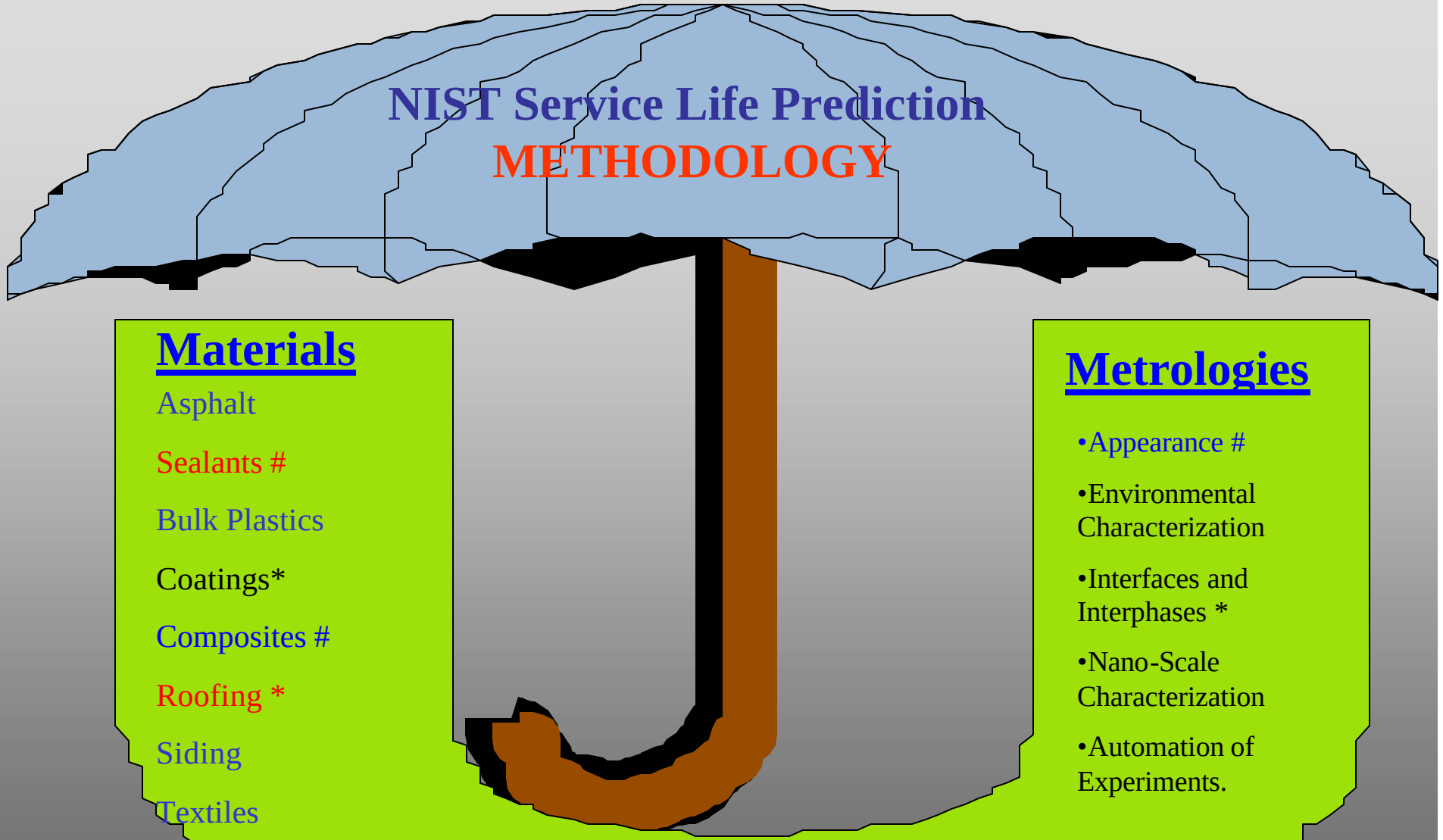
Why have we been successful with Consortia.

- We are partners.
- Involved, commitment to program.

NIST	Industry
Accept two year publication ban	Financial Commitment
Fundamental Industrial Needs	Share Information

Commit time and resources to staying in touch on time on plan

NIST Service Life Prediction
METHODOLOGY



Materials

Asphalt

Sealants #

Bulk Plastics

Coatings*

Composites #

Roofing *

Siding

Textiles

Metrologies

- Appearance #
- Environmental Characterization
- Interfaces and Interphases *
- Nano-Scale Characterization
- Automation of Experiments.

* Consortium completed, in progress, or will be established within 6 months

Consortium will most likely be established within 1 1/2 y

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Thank you

Questions?